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ARTICLE

Metal free chemoselective reduction of α-keto amides using TBAF as catalyst

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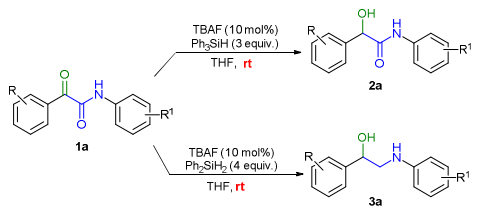
Metal and ligand free chemoselective reduction of keto group and complete reduction of the both keto and amide groups of α-keto amide with hydrosilanes using tetrabutylammoniumflouride (TBAF) as catalyst have been accomplished. This methodology affords an efficient and economic route for the synthesis of biologically important α-hydroxy amides and β-amino alcohols. The other important advantage of this TBAF catalyst is chemoselective reduction of ketones to corresponding alcohols in the presence several other sensitive functional groups.

Introduction

Chemoselective reduction is one of the most significant transformations in synthetic chemistry.[1] Metal catalyzed reduction of ketone[2] and amide[3] functional groups using hydrosilanes as reducing reagent are well established transformations in synthetic methodology. The main drawback associated with these reduction reactions is the use of expensive metals, which require inert atmosphere and ligands. It is believed that these reduction reactions generally take place through metal hydride transfer.[4] With the aid of acid,[5] base[6] or fluoride ions,[7] the hydrosilanes paved route for metal free reduction of ketones. There has been tremendous growth in the base catalyzed reduction of ester[8] and tertiary amide[9] functional groups. The serious limitation of this base catalyzed reduction is moderate selectivity and works only with tertiary amides. However, recently, much attention has been focused on the metal free reduction of secondary amides. Recently, Beller *et al*. reported metal free selective monoreduction of phthalimides and imidazolidine-2,4-diones using TBAF catalyst[10] and selective reduction of tertiary amide to amine with boronic acid catalyst.[11] Charette *et al*. reported metal free chemoselective reduction of secondary amides to amines using triflic anhydride and Et_3SiH/Hantzsch ester hydride (HEH).[12]



Scheme 1: Reduction of α-ketoamide using TBAF catalyst

Recently, reduction of aliphatic tertiary amides to amines using Cs_2CO_3 catalyst and reduction of tertiary, secondary and primary amides by $B(C_6F_5)_3$ catalyst have been reported.[13] Very recently, we have developed a chemoselective reduction of α-ketoamides using nickel catalyst.[14] As part of our on-going research towards developing metal free synthetic reactions and environmentally benign reaction conditions,[15] herein, we report, metal and ligand free chemoselective reduction of keto group of α-keto amides and complete reduction of both keto and amide groups of α-keto amide using TBAF as catalyst yielding biologically active α-hyroxy amides[16] and β-amino alcohols[17] respectively at room temperature (Scheme 1). Fluoride ion was previously been known as catalysts for the reduction of simple aldehydes, ketones and nitriles.[7e] However, to the best our knowledge, this is the first metal free fluoride ions catalyst for the selective reduction of α-keto amides using hydrosilanes.

Results and discussion

The optimization of the chemoselective reduction was initiated with 2-oxo-*N*-2-diphenylacetamide **1a** as model substrate, using polymethylhydrosilane (PMHS) as reducing agent with 5 mol% of TBAF catalyst at room temperature in THF. This reaction yielded 60% of chemoselectively reduced product α-hydroxy amide **2a** in 24 hours (Table 1, entry 1). Usage of 10 mol% of TBAF catalyst drastically increased the reactivity but the selectivity got reduced as it yielded 86% of **2a** along with 10% of β-amino alcohol **3a** (entry 2). In order to increase the chemoselectivity, various hydrosilanes were examined and the results are summarized in Table 1. Alkoxysilanehydrides such as $(EtO)_3SiH$ and $Me(EtO)_2SiH$ produced **3a** as major product with 55% and 50% yield respectively (entries 3 and 4).

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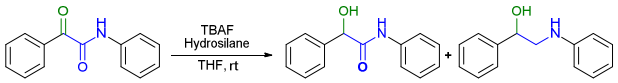
Table 1. Reduction of α-keto amide by various hydrosilanes using TBAF catalyst[a]



1a 2a 3a

Entry	Hydrosilane (equiv.)	TBAF (mol%)	Time (h)	Yield[b] (%)	
				2a	3a
1	PMHS (4)	5	24	60	-
2	PMHS (4)	10	3	86	10
3	$(EtO)_3SiH$ (4)	10	5	33	55
4	$Me(EtO)_2SiH$ (4)	10	3	38	50
5	$(Et)_3SiH$ (4)	10	24	29	-
6	Me_2ClSiH (4)	10	24	19	-
7	$MeCl_2SiH$ (4)	10	24	15	-
8	Ph_3SiH (4)	10	5	98	-
9	**Ph_3SiH (3)**	**10**	**6**	**97**	-
10	TMDSO (4)	10	8	23	72
11	**Ph_2SiH_2 (4)**	**10**	**4**	-	**95**
12	PMHS (5)	10	24	-	89
13	$(EtO)_3SiH$(5)	10	24	-	87
14	$Me(EtO)_2SiH$ (5)	10	24	-	85
15	TMDS (5)	10	24	-	92
16	Ph_2SiH_2 (4)	-	24	-	-
17	Ph_3SiH (4)	-	24	-	-

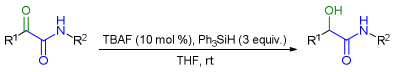
[a]Reaction condition: 0.5 mmol of **1a** in THF. [b]Isolated yield

Alkylsilanehydride such as $(Et)_3SiH$, Me_2ClSiH and $MeCl_2SiH$ are slow in reduction reaction but provided only chemoselective reduced product **2a** with poor yields 29%, 19% and 15% respectively after 24 hours (entries 5-7). Surprisingly, in the presence of 3 equivalents of Ph_3SiH, the reaction yielded 97% of exclusively **2a** after 6 hours and not even trace amount of complete reduced product **3a** was observed (entry 9). Increasing the amount of Ph_3SiH to 10 equivalents and reaction time 48 hours also did not affect the chemoselectivity. These results show that the bulkiness and reactivity of Ph_3SiH is playing a major role in the selective reduction of keto group of **1a**. In presence of $(EtO)_3SiH$, $Me(EtO)_2SiH$, tetramethyl disiloxane (TMDSO) and Ph_2SiH_2 (entries 3, 4, 10 and 11), the selectivity was reduced due to the high reactivity and small size of hydrosilanes. Me_2ClSiH and $MeCl_2SiH$ are less reactive than $(Et)_3SiH$ due to the presence of chlorine and selectively provided only **2a** with low yields (Table 1, entries 5 to 7).[18]

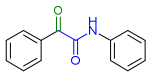
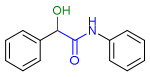
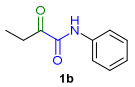
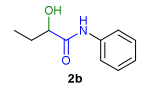
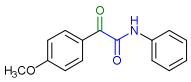
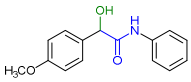
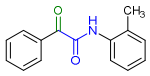
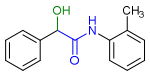
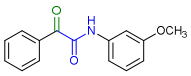
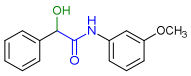
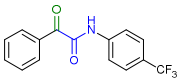
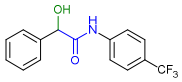
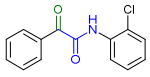
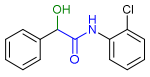
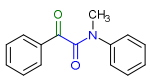
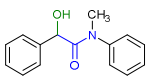
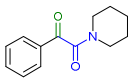
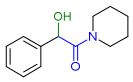
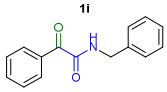
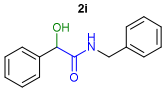
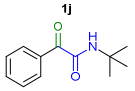
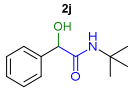
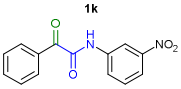
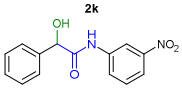
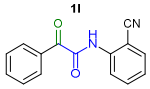
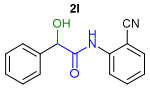
Using this optimized reaction condition (entry 9), several α-keto amides were chemoselectively reduced and the results are summarized in Table 2. Substitutions like electron withdrawing groups (**2f**, **2l** and **2m**), electron donating groups (**2d** and **2e**) on the aromatic ring attached to amide nitrogen and aliphatic amide attached ketone (**2i**, **2j** and **2k**) were well tolerated under our standard reaction condition. It is important to note that other sensitive functional groups which are prone to reduction reaction such as nitrile (**2m**), nitro (**2l**) and halo (**2g**) groups were unaffected.

After the selective reduction of keto group of α-keto amides, the focus was shifted to complete reduction to produce **3a**. In order to shift the reduction towards **3a**, strong hydrosilanes such as TMDSO and Ph_2SiH_2 were used for the reduction. As we expected, TMDSO yielded 72% of fully reduced **3a** along with **2a** as minor product (Table 1, entry 10). Using Ph_2SiH_2 as hydride transfer agent offered almost exclusive formation of **3a** in 4 hours (Table 1, entry 11). Other hydride transfer agents such as PMHS, $(EtO)_3SiH$, $Me(EtO)_2SiH$ and TMDS also provided **3a** in good yield, however the reaction time was too long (entries 12 to 15). Other fluoride sources such as CsF and KF were failed to yield **3a**. To know the role of TBAF in the reduction reactions, blank reactions were carried out only with hydrosilanes without TBAF but the reduction reaction did not take place at all (Table 1, entries 16 and 17).

Table 2. Chemoselective reduction of α-keto amide by TBAF catalyst[a]



1 2

Entry	1	2	Time (h)	Yield[b] (%)
1	 1a	 2a	6	97
2	 1b	 2b	5	92
3	 1c	 2c	5	97
4	 1d	 2d	4	98
5	 1e	 2e	5	91
6	 1f	 2f	5	88
7	 1g	 2g	6	86
8	 1h	 2h	10	89
9	 1i	 2i	8	98
10	 1j	 2j	8	86
11	 1k	 2k	10	97
12	 1l	 2l	5	98
13	 1m	 2m	7	96

[a]Reaction condition: 0.5 mmol of **1** in THF. [b]Isolated yield

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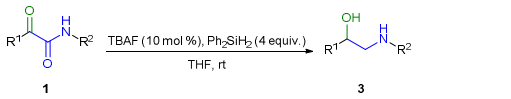
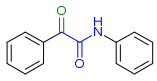
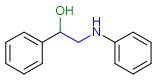
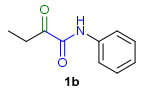
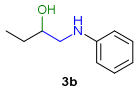
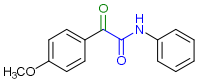
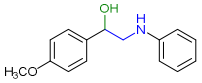
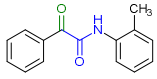
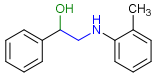
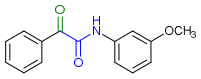
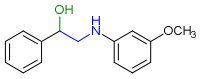
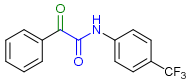
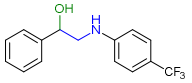
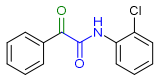
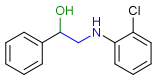
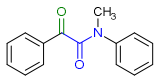
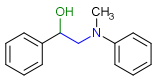
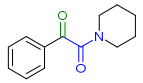
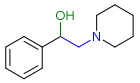
To establish the substrate scope of complete reduction, several α-keto amides were treated with Ph_2SiH_2 in presence of TBAF and the results are summarized in Table 3. Electron donating groups (**3c**, **3d** and **3e**) and electron withdrawing groups (**3f** and **3g**) did not affect the yield of the complete reduction products.[19]

Table 3. Complete reduction of α-keto amide by TBAF catalyst[a]

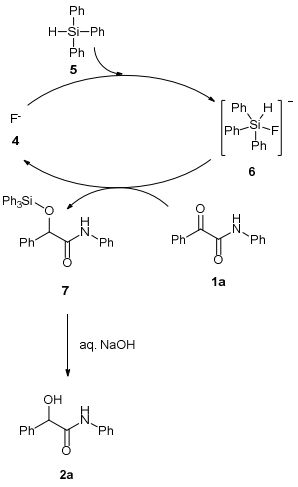


1 3

Entry	1	3	TIme (h)	Yield[b] (%)
1	 1a	 3a	4	95
2	 1b	 3b	6	92
3	 1c	 3c	4	90
4	 1d	 3d	5	87
5	 1e	 3e	5	91
6	 1f	 3f	6	88
7	 1g	 3g	7	86
8	 1h	 3h	7	82
9	 1i	 3i	6	90

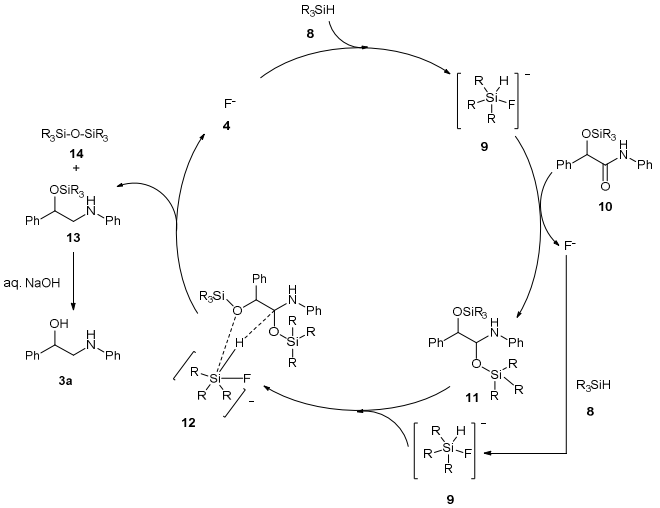
[a]Reaction condition: 0.5 mmol of **1** in THF. [b]Isolated yield

The mechanism for the TBAF-catalyzed selective reduction of α-ketoamides might be similar to the fluoride ion catalyzed hydrosilylation of ketones.[7] First, the activation of silicon atom by anionic fluorine coordination to give *penta* coordinate silicon compound[20] **6** which would then reduce selectively the ketone by transferring the hydride to the carbonyl carbon to yield the corresponding silylated amide **7**, which upon base treatment would yield **2a** (scheme 2).



Scheme 2. The plausible mechanism for the selective reduction of α-keto amides by TBAF catalyst.

The mechanism for the TBAF-catalysed complete reduction of α-ketoamides also should be similar to the TBAF-catalysed selective monoreduction of phthalimides.[10] The *penta* coordinate silicon compound **9** would reduce the amide **10** by transferring the hydride to amide to give the corresponding disilylated amine **11** (Scheme 3). Another activated hydride **9** may transfer the second hydride atom to **11** as shown in **12** to form mono silylated amine **13** then subsequent base workup should yield the complete reduced product **3a**.



Scheme 3. The plausible mechanism for the complete reduction of α-ketoamides by TBAF catalyst.

Other amides such as phenyl benzamide, methyl benzamide and *N*,2-diphenyl acetamide did not react under complete reduction condition. However, on the other hand, α-hydroxy amide **2a** and trimethylsilane protected hydroxy amide **15** react under similar reaction conditions and provided the fully reduced product in 93% and 90% respectively. In addtion, α-methoxy amide **16** did not yield even a trace amount of reduced product.

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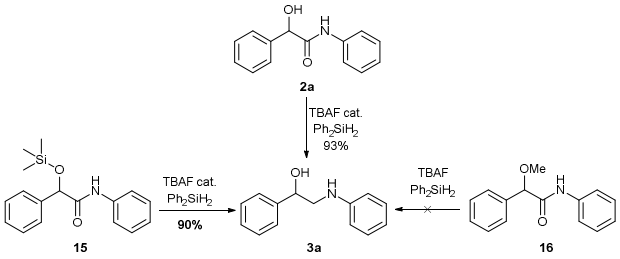
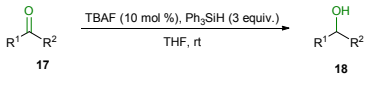
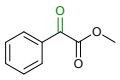
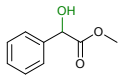
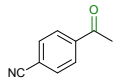
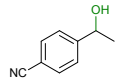
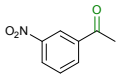
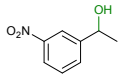
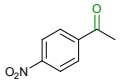
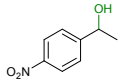
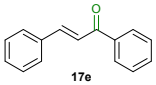
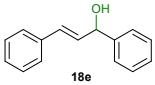
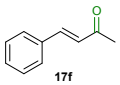
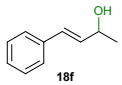
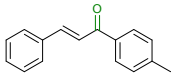
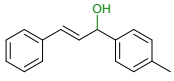
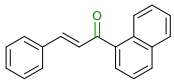
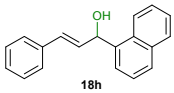
These results suggest that the α-hydroxy group of α-hydroxy amide plays a crucial role in the reduction of amide group of α-keto amide (Scheme 4).



Scheme 4

Table 4. Chemoselective reduction of ketones by TBAF catalyst[a]

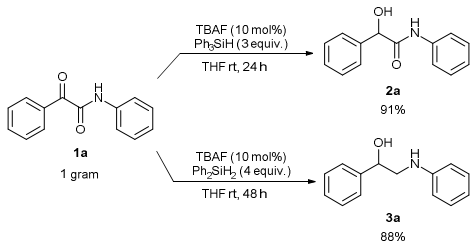


Entry	4	5	Time (h)	Yield[b] (%)
1	 17a	 18a	5	97
2	 17b	 18b	6	90
3	 17c	 18c	6	94
4	 17d	 18d	6	95
5	 17e	 18e	5	92
6	 17f	 18f	6	95
7	 17g	 18g	6	87
8	 17h	 18h	5	89

[a]Reaction condition: 0.5 mmol of **17** in THF. [b]Isolated yield

We further extended the scope of this chemoselective reduction reaction to simple ketones in the presence of several other sensitive functional groups and the results are summarized in Table 4. Various functional groups such as olefin, nitro, nitrile and ester tolerable under the catalytic system.

To examine the scalability of these metal-free reductions, the selective reduction and complete reduction of 2-oxo-*N*-2-diphenylacetamide **1a** was performed in a gram scale and the results are summarized in Scheme 5.



Scheme 5: Gram scale reaction of **1a** with TBAF

This metal free methodology affords various biologically active α-hydroxy amide and β-amino alcohols at room temperature.

Conclusions

In conclusion, we have developed an efficient transition metal and ligand free TBAF catalysed chemoselective and complete reduction of α-keto amides using appropriate hydrosilanes at room temperature. The present metal-free reduction method have several advantages including short reaction times, high yields, reaction at room temperature, chemoselective and complete reductions using appropriate hydrosilanes. The important advantage of using TBAF catalyst is chemoselective reduction of ketones to corresponding alcohols in the presence several other sensitive functional groups.

Experimental Section

General Considerations

Hydrosilanes and TBAF (1.0 M solution in THF) were purchased from Sigma Aldrich chemical company. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F254 pre-coated plates (0.25 mm) and visualized by UV fluorescence quenching. Silica gel for column chromatography (particle size 100-200 mesh) was purchased from SRL India. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz instrument. ^{1}H NMR spectra were reported relative to residual $CHCl_3$ (δ 7.26 ppm) or DMSO-d_6 (δ 2.50 ppm). ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.16 ppm) or DMSO-d_6 (δ 39.52 ppm). FTIR spectra were recorded on a JASCO spectrometer and are reported in frequency of absorption (cm^{-1}). High resolution mass spectra (HRMS) were recorded on Q-Tof Micro mass spectrometer.

General experimental procedure for synthesis of α-keto amides

Thionyl chloride (0.3 mL, 4 mmol) was added drop wise to a stirred mixture of benzyl formic acid (0.300 g, 2 mmol) and Et_3N (0.5 mL, 4 mmol) in CH_2Cl_2 (10 mL) at 0 ^{o}C under

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nitrogen atmosphere. The stirring was continued for 20 min. and then a suspension of corresponding amine (2 mmol) in CH_2Cl_2 (10 mL) was added slowly to the reaction mixture at 0 ^oC under nitrogen flow. The stirring was continued in the room temperature and the completion of the reaction was monitored by TLC. A saturated aqueous solution of $NaHCO_3$ (20 mL) was slowly added to the reaction mixture. The organic layer was separated, washed with water (3 × 15 mL) and evaporated under reduced pressure. The solid residue was purified by silica gel column chromatography.

General experimental procedure for chemoselective reduction of α-keto amides using TBAF catalyst

A mixture of TBAF (1M solution in THF, 50 µL, 0.05 mmol) and α-keto amide (0.5 mmol) in 1.5 mL of distilled THF was taken in a reaction tube. The reaction mixture was stirred at room temperature for 10 min. Then Ph_3SiH (390 mg, 1.5 mmol) was added to the reaction mixture. After that, the resulting reaction mixture was stirred at room temperature under open air. The progress of the reaction was monitored by TLC. After complete disappearance of substrate, 10 mL of 2N aq. NaOH was added to the reaction and the resulting mixture was stirred for 10 min. The reaction mixture was extracted with ethyl acetate (2x10 mL). The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered off and the solvent was removed under reduced pressure. The resulting residue was purified by silica gel column chromatography (eluents: hexanes-ethyl acetate, 80:20) to obtain pure α-hydrxoy amide.

General experimental procedure for complete reduction of α-keto amides using TBAF catalyst

A mixture of TBAF (1M solution in THF, 50 µL, 0.05 mmol) and α-keto amide (0.5 mmol) in 1.5 mL of dry THF was taken in a reaction tube at 0 ^oC. The reaction mixture was stirred at 0 ^oC temperature for 10 min. Then Ph_2SiH_2 (370 µL, 2 mmol) was slowly added drop wise to the reaction mixture at 0 ^oC under nitrogen atmosphere. After that, the reaction tube was closed with glass stopper under nitrogen flow and the resulting reaction mixture was stirred at room temperature. The progress of the reaction was monitored by TLC. After complete disappearance of substrate, 5 mL of 2N aq. NaOH was added to the reaction and the resulting mixture was stirred for 10 min. The reaction mixture was extracted with ethyl acetate (2x10 mL). The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered off and the solvent was removed under reduced pressure. The resulting residue was purified by neutral aluminium oxide column chromatography (eluents: hexanes-ethyl acetate, 90:10) to obtain pure β-amino alcohol.

2-Hydroxy-*N*, 2-diphenylacetamide (2a):
Colourless solid, mp = 152-153 ^oC (lit.[14] mp = 152-153 ^oC); R_f 0.56; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.19 (bs, 1H), 7.51 (d, J =7.6 Hz, 2H), 7.47 (d, J = 6.8 Hz, 2H), 7.42-7.34 (m, 3H), 7.31 (t, J = 7.2 Hz, 2H), 7.12 (t, J = 7.2 Hz, 1H), 5.15 (d, J = 2.0 Hz, 1H), 3.57 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.1, 139.1, 137.2, 129.2, 129.2, 129.1, 129.0, 127.0, 119.9, 74.8; IR (KBr) 3232, 1666, 1660, 1651, 1600, 1066 cm^{-1}; HRMS (*m/z*): $[M+Na]^+$ calcd for $C_{14}H_{13}NO_2Na_1$, 250.0844; found, 250.0846.

2-Hydroxy-*N*-phenylbutanamide (2b):
Colorless solid, mp = 129-130 ^oC (lit.[14] mp = 129-130 ^oC); R_f 0.30; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.42 (bs, 1H), 7.56 (dd, J = 8.6, 1.2 Hz, 2H), 7.33 (t, J =7.6 Hz, 2H), 7.12 (t, J = 8.4 Hz, 1H), 4.24-4.18 (m, 1H), 2.81 (d, J = 4.8 Hz, 1H), 2.03-1.91 (m, 1H), 1.86-1.74 (m, 1H), 1.03 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 137.3, 129.2, 124.7, 119.9, 73.7, 28.0, 9.3; IR (KBr) 3400, 1646, 1600, 1528, 1443, 1119 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{10}H_{14}NO_2$, 180.1025; found, 180.1031.

2-Hydroxy-2-(4-methoxyphenyl)-N-phenylacetamide (2c):
Colorless solid, mp = 94-95 ^oC (lit.[14] mp = 94-95 ^oC); R_f 0.32; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.17 (bs, 1H), 7.52 (d, J = 7.6 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 7.31(t, J = 7.6 Hz, 2H), 7.12 (t, J = 7.2 Hz, 1H), 6.90 (d, J = 8.4 Hz, 2H), 5.11 (s. 1H), 3.80 (s, 3H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.4, 160.2, 137.2, 131.3, 129.2, 128.5 124.8, 119.9, 114.5, 74.4, 55.5; IR (KBr) 3271, 1644, 1594, 1532, 1442, 1065 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{16}NO_3$, 258.1130; found, 258.1133.

2-Hydroxy-2-phenyl-*N*-(o-tolyl)acetamide (2d):
Colorless solid, mp = 85-86 ^oC; R_f 0.36; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.20 (bs, 1H), 7.82 (d, J = 8.4 Hz, 1H), 7.44 (dd, J = 7.6, 2.0 Hz, 2H), 7.32-7.39 (m, 3H), 7.13-7.19 (m, 2H), 7.06 (td, J = 6.4,1.2 Hz, 1H), 5.08 (s, 1H), 4.16 (s, 1H), 2.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 139.3, 135.3, 135.0,130.6, 128.8 127.8, 126.9, 126.8, 125.4, 122.3, 74.7, 17.5; IR (KBr) 3319, 1668, 1588, 1532, 1493, 1064 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{16}NO_2$, 242.1181; found, 242.1180.

2-Hydroxy-N-(3-methoxyphenyl)-2-phenylacetamide (2e):
Colorless solid, mp = 132-133 ^oC; R_f 0.31; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 9.87 (bs, 1H), 7.50 (d, J = 7.2 Hz 2H), 7.40 (t, J = 1.6 Hz, 1H), 7.35(t, J = 6.8 Hz, 2H), 7.26-7.31 (m, 2H), 7.18 (t, J = 8.0 Hz, 1H), 6.62 (dd, J = 8.2, 1.6 Hz, 1H), 6.42 (d, J = 4.8 Hz, 1H), 5.08 (d, J = 4.4 Hz, 1H), 3.70 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 159.5, 140.8, 139.7, 129.4, 128.1, 127.6, 126.6, 111.9, 109.1, 105.3, 74.0, 55.0; IR (KBr) 3409, 3327, 1654, 1607, 1561, 1496, 1053 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{16}NO_3$, 258.1130; found, 258.1138.

2-Hydroxy-2-phenyl-N-(4-(CF_3)phenylacetamide (2f):
Colorless solid, mp = 176-177 ^oC; R_f 0.40; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 10.33 (bs, 1H), 7.96 (d, J = 8.4 Hz, 2H), 7.68 (d, J = 8.4 Hz, 2H), 7.54 (d, J = 7.2 Hz, 2H), 7.38 (t, J = 7.6 Hz, 2H), 7.29-7.34 (m, 1H),

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6.55 (d, J = 4.4 Hz, 1H), 5.17 (d, J = 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.9, 142.2, 140.5, 134.5, 128.1, 128.0, 127.7, 126.6, 125.9 (q, J = 3.6 Hz, CF_3), 119.7, 74.1; IR (KBr) 3235, 1666, 1614, 1604, 1556, 1072 cm^{-1}; HRMS (m/z): $[M+Na]^+$ calcd for $C_{15}H_{12}NO_2F_3Na_1$, 318.0718; found, 318.0723.

***N*-(2-chlorophenyl)-2-hydroxy-2-phenylacetamide (2g):**
Colorless solid, mp = 96-97 °C; R_f 0.36; (hexanes : ethyl acetate, 70:30 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.64 (bs, 1H), 8.09 (dd, J = 8.0, 1.6 Hz, 1H), 7.53 (dd, J = 8.2, 1.6 Hz, 1H), 7.49 (d, J = 7.8 Hz, 2H), 7.28-7.40 (m, 4H), 7.16 (td, J = 7.8, 1.6 Hz, 1H) 6.94 (d, J = 4.0 Hz, 1H), 5.18 (d, J = 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 140.4, 134.1, 129.4, 128.2, 127.8, 127.8, 126.7, 125.5, 123.8, 122.2, 73.6; IR (KBr) 3289, 1671, 1596, 1590, 1553, 1436, 1058 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{14}H_{13}NO_2Cl$, 262.0635; found, 262.0648.

2-Hydroxy-N-methyl-N,2-diphenylacetamide (2h):
Colorless solid, mp = 89-90 °C (lit.[14] mp = 89-90 °C) ; R_f 0.40; (hexanes : ethyl acetate, 70:30 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-7.13 (m, 3H), 7.14-7.04 (m, 3H), 6.76- 6.74 (m, 4H), 4.95 (s, 1H), 3.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.9, 141.5, 139.3, 129.6, 128.4, 128.1, 128.0, 127.3, 71.7, 38.4; IR (KBr) 3433, 1652, 1591, 1490, 1447, 1084 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{15}H_{16}NO_2$, 242.1181; found, 242.1192.

2-Hydroxy-2-phenyl-1-(piperidin-1-yl)ethan-1-one (2i):
Colorless solid, mp = 78-79 °C (lit.[21] 77 °C) ; R_f 0.55; (hexanes : ethyl acetate, 70:30 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.28 (m, 5H), 5.20 (s, 1H), 3.81-3.73 (m, 1H), 3.49-3.43 (m, 1H), 3.17 (t, J = 4.4 Hz, 2H), 1.62-1.41 (m, 5H), 1.36-1.27 (m, 1H), 0.95-0.85 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 139.8, 129.1, 128.5, 127.5, 71.5, 45.9, 44.1, 25.4, 25.2, 24.2; IR (KBr) 3223, 1625, 1449, 1361, 1264, 1081 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{13}H_{18}NO_2$, 220.1338; found, 220.1340.

N-Benzyl-2-hydroxy-2-phenylacetamide (2j):
Colorless solid, mp = 133-134 °C (lit.[14] mp = 133-134 °C); R_f 0.35; (hexanes : ethyl acetate, 80:20 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.32 (m, 5H),7.32-7.25 (m, 3H), 7.18 (dd, J = 6.0, 2.0 Hz, 2H), 6.47 (bs, 1H), 5.06 (d, J = 2.8 Hz, 1H), 4.49-4.36 (m, 2H), 3.68 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.2, 139.5, 137.8, 129.0, 128.9, 127.7, 127.0, 74.4, 43.7; IR (KBr) 3232, 1732, 1625, 1535, 1448, 1060 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{15}H_{16}NO_2$, 242.1171; found, 242.1181.

N-tert-Butyl-2-hydroxy-2-phenylacetamide (2k):
Colorless solid, mp = 103-104 °C (lit.[14] mp = 103-104 °C); R_f 0.31; (hexanes : ethyl acetate, 70:30 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.39 (m, 5H), 5.83 (bs, 1H), 4.89 (s, 1H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.4, 140.0, 129.0, 128.7, 127.0, 74.4, 51.7, 28.8; IR (KBr) 3225, 1647, 1539, 1479, 1064 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{12}H_{18}NO_2$, 208.1338; found, 208.1339.

2-Hydroxy-N-(3-nitrophenyl)-2-phenylacetamide (2l):
Colorless solid, mp = 142-143 °C; R_f 0.42; (hexanes : ethyl acetate, 70:30 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.50 (bs, 1H), 8.78 (s, 1H), 8.14 (d, J = 8.0 Hz, 1H), 7.92 (dd, J = 8.2, 1.2 Hz, 1H), 7.60 (t, J = 8.0 Hz, 1H), 7.55 (d, J = 7.6 Hz, 2H), 7.38 (t, J = 7.2 Hz, 2H), 7.32 (t, J = 7.2 Hz, 1H), 6.59 (s, 1H), 5.17 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.1, 147.9, 140.5, 139.8, 130.0, 128.2, 127.8, 126.6, 125.9, 118.1, 113.9, 74.1; IR (KBr) 3213, 1659, 1620, 1603, 1550, 1067 cm^{-1}; HRMS (m/z): $[M+Na]^+$ calcd for $C_{14}H_{12}N_2O_4Na_1$, 295.0695; found, 295.0690.

N-(2-cyanophenyl)-2-hydroxy-2-phenylacetamide (2m):
Colorless solid, mp = 128-129 °C; R_f 0.37; (hexanes : ethyl acetate, 70:30 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.12 (bs, 1H), 7.84 (d, J = 8.0 Hz, 2H), 7.66-7.73 (m, 1H), 7.54 (d, J = 7.2 Hz, 2H), 7.29-7.42 (m, 4H), 6.82 (d, J = 4.4 Hz, 1H), 5.20 (d, J = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.4, 140.4, 139.8, 133.9, 133.0, 128.2, 127.8, 126.8, 125.4, 123.7, 166.5, 105.8, 73.6; IR (KBr) 3311, 2234, 1687, 1601, 1577, 1058 cm^{-1}; HRMS (m/z): $[M+Na]^+$ calcd for $C_{15}H_{12}N_2O_2Na_1$, 275.0796; found, 275.0801.

1-Phenyl-2-(phenylamino)ethanol (3a):
Colourless oily liquid; R_f 0.36; (hexanes : ethyl acetate, 80:20 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.36 (m, 4H), 7.35-7.31 (m, 1H), 7.24-7.18 (m, 2H), 6.82-6.77 (m, 1H), 6.75 (dd, J = 8.6, 1.2 Hz, 2H), 4.96 (dd, J = 8.6, 4.0 Hz, 1H), 3.45 (dd, J = 13.0, 3.6 Hz, 1H), 3.32 (dd, J = 13.0, 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.3, 142.0, 129.5, 128.8, 128.2, 126.0, 119.0, 114.2, 72.4, 52.5; IR (Neat) 3402, 1708, 1600, 1503, 1055 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{14}H_{16}NO$, 214.1232; found, 214.1230.

1-(Phenylamino)butan-2-ol (3b):
Colourless oily liquid; R_f 0.40; (hexanes : ethyl acetate, 80:20 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77-7.72 (m, 2H), 7.22-7.15 (m, 2H), 6.68 (dd, J = 8.4, 1.2 Hz, 2H), 3.82-3.74 (m, 1H), 3.28 (dd, J = 12.8, 3.2 Hz, 1H), 3.02 (dd, J = 12.8, 8.5 Hz, 1H), 1.64-1.51 (m, 2H), 1.02 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 129.5, 118.3, 113.6, 71.8, 50.2, 28.1, 10.1; IR (Neat) 3623, 3549, 1740, 1605, 1464, 1047 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{10}H_{16}NO$, 166.1232; found, 166.1228.

1-(4-Methoxyphenyl)-2-(phenylamino)ethan-1-ol (3c):
Pale yellow oily liquid; R_f 0.37; (hexanes : ethyl acetate, 80:20 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.31 (m, 2H), 7.23-7.17 (m, 2H), 6.95-6.90 (m, 2H), 6.76 (t, J = 7.2 Hz, 1H), 6.69 (dd, J = 8.6, 1.2 Hz, 2H), 4.89 (dd, J = 8.4, 4.0 Hz, 1H), 3.82 (s, 3H), 3.40 (dd, J = 12.8, 4.0 Hz, 1H), 3.31 (dd, J = 13.0, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.6, 147.9, 134.2, 129.5, 127.3, 118.4, 114.2, 113.7, 72.2, 55.5, 52.0; IR (Neat)

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3646, 1663, 1615, 1601, 1049 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{18}NO_2$, 244.1338; found, 244.1330

1-Phenyl-2-(o-tolylamino)ethan-1-ol (3d):
Colourless oily liquid; R_f 0.35; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.76-7.38 (m, 4H), 7.36-7.31 (m, 1H), 7.17-7.12 (m, 1H), 7.10-7.06 (m, 1H), 6.74-6.68 (m, 2H), 4.98 (dd, *J* = 8.4, 4.0 Hz, 1H), 3.48 (dd, *J* = 12.8, 4.0 Hz, 1H) 3.36 (dd, *J* = 13.0, 8.4 Hz, 1H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.9, 142.2, 130.4, 128.8, 128.2, 127.3, 126.0, 122.9, 117.8, 110.5, 72.6, 51.8, 17.6; IR (Neat) 3411, 1602, 1511, 1453, 1051 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{18}NO$, 228.1388; found, 228.1397.

2-((3-Methoxyphenyl)amino)-1-phenylethan-1-ol (3e):
Pale yellow oily liqued; R_f 0.36; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.41-7.32 (m, 5H), 7.11 (t, *J* = 8.0 Hz, 1H), 7.35-6.31 (m, 1H), 6.31-6.26 (m, 1H), 6.23 (t, *J* = 2.0 Hz, 1H), 4.89 (dd, *J* = 8.6, 4.0 Hz, 1H), 3.77 (s, 3H), 3.39 (dd, *J* = 13.2, 4.0 Hz, 1H), 3.27 (dd, *J* = 13.8, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.1, 149.5, 142.2, 130.2, 128.8, 128.2, 126.0, 106.6, 103.4, 99.7, 72.7, 55.3, 51.9; IR (Neat) 3465, 1613, 1456, 1411, 1049 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{18}NO_2$, 244.1338; found, 244.1328.

1-Phenyl-2-((4-(CF_3)phenyl)amino)ethan-1-ol (3f):
Pale yellow oily liqued; R_f 0.39; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.44-7.33 (m, 7H), 6.67 (d, *J* = 8.4 Hz, 2H), 4.93 (dd, *J* = 8.4, 4.0 Hz, 1H), 3.45 (dd, *J* = 13.2, 4.0 Hz, 1H), 3.35 (dd, *J* = 13.2, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.4, 141.8, 134.2, 128.9, 128.4, 127.8, 126.8 (q, *J* = 3.5 Hz, CF_3), 126.0, 112.6, 72.7, 51.1; IR (Neat) 3411, 1663, 1616, 1536, 1054 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{15}F_3NO$, 282.1106; found, 282.1113.

2-((2-Chlorophenyl)amino)-1-phenylethan-1-ol (3g):
Colorless oily liquid; R_f 0.38; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.45-7.38 (m, 4H), 7.34 (tt, *J* = 5.6, 1.2 Hz, 1H), 7.28 (dd, *J* = 6.4, 1.2 Hz, 1H), 7.18-7.13 (m, 1H), 6.79 (dd, *J* = 6.4, 0.8 Hz, 1H), 6.70 (td, *J* = 6.0, 1.2 Hz, 1H), 5.00 (dd, *J* = 6.8, 3.2 Hz, 1H), 3.48 (dd, *J* = 10.4, 3.2 Hz, 1H) 3.39 (dd, *J* = 10.4, 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.4, 141.8, 129.5, 128.9, 128.3, 128.0, 126.0, 120.3, 118.6, 112.4, 72.5, 51.9; IR (Neat) 3411, 1718, 1663, 1593, 1062 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{15}ClNO$, 248.0842; found, 248.0852.

2-(Methyl(phenyl)amino)-1-phenylethanol (3h):
Colorless oily liqued; R_f 0.62; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.39-7.30 (m, 4H), 7.29-7.19 (m, 3H), 6.81 (d, *J* = 8.0 Hz, 2H), 6.74 (tt, *J* = 7.2, 0.8 Hz, 1H), 4.93 (dd, *J* = 8.6, 4.4 Hz, 1H), 3.46 (dd, *J* = 14.6, 8.8 Hz, 1H), 3.38 (dd, *J* = 14.8, 4.4 Hz, 1H), 2.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.0, 142.1, 129.4, 128.7, 127.9, 126.0, 117.7, 113.4, 71.8, 62.1, 39.6; IR (Neat) 3404, 1453, 1374, 1047 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{18}NO$, 228.1388; found, 228.1386.

1-Phenyl-2-(piperidin-1-yl)ethan-1-ol (3i):
Colorless oily liqued; R_f 0.54; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.31-7.23 (m, 4H), 7.18 (tt, *J* = 5.6, 1.2 Hz, 1H), 4.65 (dd, *J* = 8.4, 2.8 Hz, 1H), 2.62 (bs, 2H) 2.41 (dd, *J* = 9.8, 2.8 Hz, 1H), 2.32 (dd, *J* = 10.0, 8.4 Hz, 3H), 1.61-1.48 (m, 4H), 1.40 (q, *J* = 4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.6, 128.4, 127.5, 126.0, 68.8, 67.1, 54.6, 26.2, 24.4; IR (Neat) 3143, 1445, 1326, 1272, 1036 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_{13}H_{20}NO$, 206.1545; found, 206.1555.

Methyl 2-hydroxy-2-phenylacetate (18a):
Colorless oily liquid; R_f 0.58; (hexanes : ethyl acetate, 70:30 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.44-7.40 (m, 2H), 7.39-7.32 (m, 3H), 5.18 (s, 1H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.2, 138.4, 128.7, 128.6, 126.7, 73.0, 53.0; IR (KBr) 3459, 1740, 1636, 1493, 1440, 1093 cm^{-1}; HRMS (*m/z*): $[M+Na]^+$ calcd for $C_9H_{10}O_3Na_1$, 189.0528; found, 189.0524.

4-(1-Hydroxyethyl)benzonitrile (18b):
Colorless oily liquid; R_f 0.42; (hexanes : ethyl acetate, 70:30 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.61 (dt, *J* = 8.4, 1.6 Hz, 2H), 7.47 (dd, *J* = 8.4, 0.4 Hz, 2H), 4.94 (q, *J* = 6.4 Hz, 1H), 1.48 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.3, 132.4, 126.2, 119.0, 111.1, 69.7, 25.5; IR (Neat) 3417, 2229, 1609, 1504, 1407, 1090 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_9H_{10}NO_2$, 148.0762; found, 148.0768.

1-(3-Nitrophenyl)ethan-1-ol (18c):
Colorless oily liquid; R_f 0.48; (hexanes : ethyl acetate, 70:30 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.18 (t, *J* = 2.0 Hz, 1H), 8.05 (ddd, *J* = 7.6 Hz, 1H), 7.66 (d, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 7.6 Hz, 1H) 4.97 (q, *J* = 6.4 Hz, 1H), 2.78 (bs, 1H) , 1.48 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 148.0, 131.7, 129.5, 122.3, 120.4, 69.3, 25.4; IR (KBr) 3371, 1530, 1481, 1448, 1074 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_8H_{10}NO_3$, 168.0661; found, 168.0660.

1-(4-Nitrophenyl)ethan-1-ol (18d):
Colorless oily liqued; R_f 0.46; (hexanes : ethyl acetate, 70:30 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.18 (d, *J* = 8.8 Hz, 2H), 7.53 (d, *J* = 8.8 Hz, 2H), 5.01 (q, *J* = 6.8 Hz, 1H), 2.06 (bs, 1H), 1.51 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.2, 147.3, 126.2, 123.9, 69.6, 25.6; IR (KBr) 3387, 1602, 1518, 1451, 1086 cm^{-1}; HRMS (*m/z*): $[M+H]^+$ calcd for $C_8H_{10}NO_3$, 168.0661; found, 168.0665.

(*E*)-1,3-diphenylprop-2-en-1-ol (18e):
Pale yellow solid; mp = 57-58 ^oC, R_f 0.31; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 7.6 Hz, 2H), 7.43-7.38 (m, 4H), 7.37-7.31 (m, 3H), 7.31-7.25 (m, 1H), 6.72 (d, *J* = 16.0 Hz, 2H), 6.42 (dd, *J* = 16.0, 6.4 Hz, 1H), 5.41 (d, *J* = 6.4 Hz, 1H), 2.24 (bs, 1H); ^{13}C NMR (100

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MHz, $CDCl_3$): δ 142.9, 136.7, 131.7, 130.7, 128.7, 128.6, 127.9, 127.2, 126.7, 126.5, 75.2; IR (KBr) 3406, 1645, 1604, 1492, 1445 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{15}H_{15}O$, 211.1123; found, 211.1125.

(*E*)-4-phenylbut-3-en-2-ol (18f):
Colorless oily liquid, R_f 0.28; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.33-7.29 (m, 2H), 7.24 (td, *J* = 6.8, 2.0 Hz, 2H), 7.19-7.14 (m, 1H), 6.49 (d, *J* = 16.0 Hz, 1H), 6.19 (dd, *J* = 16.0, 6.4 Hz, 1H) 4.46-4.38 (m, 1H), 1.62 (bs, 1H), 1.30 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.8, 133.7, 129.5, 128.7, 127.8, 126.6, 69.1, 23.5; IR (Neat) 3354, 2973, 1646, 1494, 1450, 1062 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{10}H_{13}O$, 149.0966; found, 149.0967.

(*E*)-3-phenyl-1-(p-tolyl)prop-2-en-1-ol (18g):
Pale yellow oily liquid; R_f 0.34; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.2 Hz, 2H), 7.35 (t, *J* = 8.0 Hz, 4H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 2H), 6.72 (d, *J* = 16.0 Hz, 1H), 6.42 (dd, *J* = 16.0, 6.4 Hz, 1H), 5.39 (d, *J* = 6.0 Hz, 1H), 2.40 (s, 3H), 2.22 (bs, 1H); ^{13}C NMR (100 MHz, CDCl3): δ 140.0, 137.6, 136.7, 131.8, 130.4. 129.4, 128.7, 127.8, 126.7, 126.5, 21.2; IR (Neat) 3453, 1605, 1505, 1452, 1039 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{16}H_{17}O$, 225.1279; found, 225.1280.

(*E*)-1-(naphthalen-1-yl)-3-phenylprop-2-en-1-ol (18h):
Yellow oily liquid; R_f 0.31; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, $CDCl_3$): δ 8.04-8.00 (m, 1H), 7.68 (dd, *J* = 6.8, 2.8 Hz, 1H), 7.61 (d, *J* = 7.2 Hz, 1H), 7.33-7.25 (m, 3H), 7.19-7.15 (m, 2H), 7.12-7.06 (m, 2H), 7.05-7.01 (m, 1H) 6.57 (d, *J* = 16.0, 1H), 6.37 (dd, *J* = 16.0, 6.0 Hz, 1H), 5.88 (d, *J* = 5.6 Hz, 1H), 2.10 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.4, 136.7, 134.1, 131.2, 131.1, 130.8, 129.0, 128.7, 127.9, 126.7, 126.3, 125.8, 125.6, 124.1, 123.9, 72.2; IR (Neat) 3595, 2252, 1643, 1600, 1501, 1454 cm^{-1}; HRMS (m/z): $[M+H]^+$ calcd for $C_{19}H_{17}O$, 261.1279; found, 261.1281.

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Notes and references

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